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64 Electrodeposition coating composition comprising crosslinked microparticles.

67) An electrodeposition coating composition is described comprising:

(a) an aqueous dispersion of a water-dispersible, electrically-depositable, at least partially neutralized cationic resin, and

(b) polymer microparticles, said polymer particles being prepared by:

(1) blending an acid-neutralized tertiary amino-functional acrylic polymer and a polyepoxide,

(2) dispersing the blended mixture in an aqueous medium to form a dispersion of microparticles of the blended mixture, and

(3) heating the dispersion to crosslink the acrylic and polyepoxide in the microparticles.

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This invention relates to electrodepositions coating compositions, and in particular to such compositions including crosslinked microparticles.

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Electrodeposition coating, or electrocoating, is widely used in the artfor the application of polymer coatings to metal substrates. Electrodeposition baths usually comprise a principal film-forming resin, such as an acrylic or epoxy resin, with ionic groups that can be salted so that the resin can be dispersed or dissolved in an aqueous bath. Pigments (dispersed in resin pastes), dyes, flow control agents, and other additives are often included in the electrocoat bath.

For automotive or industrial applications where hard electrocoat films are desired, the bath also includes a blocked crosslinking agent that unblocks under appropriate conditions (e.g., with the application of heat) to react with functional groups on the principal resin and thus cure the coating.

One of the advantages of electrodeposition coating compositions and processes is that the coating composition can be applied to a variety of metallic substrates regardless of shape or configuration. This is especially advantageous when the coating is applied as an anticorrosive coating onto a substrate having a number of irregular surfaces such as a motor vehicle body. In order to maximize an electrodeposition coating's anticorrosion effectiveness at is important that the coating form a contiguous layer over all portions of the metallic substrates some and decays easily are a large as large and the metallic substrates some and decays easily are a large as large and as a large as large as large as a large as large

Two criteria for measuring the effectiveness of an electrodeposition coating for covering all portions of the substrate are throwpower and edge coverage. Throwpower measures the effectiveness of an electrodeposition coating at covering recessed or interior areas of a metal substrate. Edge coverage measures the effectiveness of an electrodeposition coating at covering the edges of a metallic substrate. Good throwpower and edge coverage are important in order to maximize an electrodeposition coating's anticorrosion effectiveness.

Electrodeposition coatings must often satisfy a number of other criteria as well. A high degree of smoothness is often desirable. For example, when the electrodeposition coating serves as a primer for a high-gloss topcoat, the primer layer must be very smooth in order for the topcoat to have a satisfactory appearance. It is also advantageous to exhibit stability over a range of ph.

and edge coverage, without compromising overal corrosion protection and smoothness.

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vertical to (b) polymer microparticles; said polymer particles being prepared by: vertices assume a substitute of

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 - 2 (2) dispersing the blended mixture in an aqueous medium to form a dispersion of microparticles of the blended mixture, and

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and however, (3) heating the dispersion to crosslink the acrylic and polyepoxide in the microparticles. Additionally

When used in an electrodeposition process, the coating composition of the invention provides a smooth, contiguous coating over a variety of portions of the metal substrate, including recessed areas and edges. The coating composition is thus highly effective as an anticorrosive primer coating for metal substrates, particularly for motor vehicle bodies.

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The microparticles used in the practice of the present invention are prepared from a neutralized tertiary amine-functional acrylic polymer and a polyepoxide. The tertiary amine-functional acrylic polymer can be prepared from one or more acrylic monomers containing tertiary amine groups in the ester portion of the molecule and one or more other copolymerizable ethylenically-unsaturated monomers. Tertiary amino group-containing acrylic monomers are well-known in the art and include, for example, dimethylamino ethyl methaccylate and dimethylamino ethyl acrylate. Copolymerizable ethylenically-unsaturated monomers are also well-known in the art. Such monomers priferably do not/containany groups that would be riactive with amine. They include alkyl esters of acrylic or methacrylic acid, e.g., ethyl acrylate, butyl acrylate, 2-ethylhexyl acrylate, butyl me-

thacrylate, isodecyl methacrylate, hydroxyethyl methacrylate, hydroxypropyl acrylate, and the like; and vinyl monomers such as styrene, vinyl toluene, and the like.

Alternatively, the tertiary amine-functional acrylic polymer can be prepared by first forming an acrylic polymer backbone having side groups that can be reacted with another compound so as to attack a tertiary amino group onto the backbon . This can be accomplished, for example, by incorporating glycidyl methacrylate into an acrylic polymer, and then reacting the oxirane side groups with a secondary/amin'es it is among the

The tertiary amine-functional acrylic polymer preferably has a number average molecular weight of from 43000 to 80,000, and more preferably of from 40,000 to 25,000.5 he polymer preferably has an equivalent weight per tertiary nitrogen of 400 to 1500, and more preferably of 750 to 1500 hands are Elizentonius and

Among the polyepoxides that can be used are epoxy condensation polymers (e.g., polyglycidyl ethers of alcohols and phenois), which are preferred, epoxy-containing acrylic polymers, and certain polyepoxide monomers and oligomers. le electrocoutesie el

The epoxy condensation polymers that are used are polyepoxides; that is, those having a 1,2-epoxy equivalency greater than 1, preferably greater than 1 and up to about 3.0 in one preferred embodiment, the polyepoxide is a diepoxide, and thus has at 1,2-epoxy equivalency of 2. Examples of such epoxides are polyglycidyl ethers of polyhydric phenols and of aliphatic alcohols. These polyepoxides can be produced by etherification of the polyflydric phenol or aliphatic alcohol with an epihalohydrin; such as epichlorohydrin; in the presence of . Tá ta**dac**ous when ath metur connective concentration is the logarity and name sucapears the

ுல்கு இதுக்கூறிய விக்கிய polyphenols are 2,2-bis(4-hydroxyphenyl)propane (bisphenol A), வி.நி.நி.நி. நி. முர்கு முர்கள் 20 phenyl)ethane, and 2-methyl-\$,1-bis(4-hydroxyphenyl)propane. Examples of suitable aliphatic-alcohols are ethylene glycol, diethylene glycol, 1,2-propylene glycol, and 1,4-butylene glycol. Also, cycloaliphatic polyols isuch as 1,2-cyclohexanediol; 1,4-cyclohexanediol; 1,2-bis(hydroxymethyl) cyclohexane, and hydrogenated bi-्र प्रेम्बारहोत यह से troypower and edge coverage. Tarowpover maasing stra she**bearredooslainasi Alfonedds** कि

Besides the epoxy-containing polymers described above certain polyepoxide monomers and oligomers 25 💮 can also be used. Examples of these materials are those containing the cyclohexane oxide mojety. These polyepoxides are of relatively low molecular weight and of relatively high reactivity, thus enabling the formation of the high solids coating compositions with excellent cure response. The polyepoxides should have an average 1,2in process are dispositely of greater than one. The preferred polyepoxides are disposides; that is, having a 1,2-epoxy led the gainer layer must be very a nooth beater for the properties have a satisf**owtho yonelsviupe**. It is

The epoxy-containing acrylic polymer is a copolymer of an ethylenically unsaturated ponomer having at welleast one epoxy group and at least one polymerizable ethylenidally unsaturated monomer that is free of epoxy groups. sesone community for interesting areas and larger problems and the energy acceptance of

Examples of ethylenically unsaturated monomers containing epoxy groups are those containing 1,2-epoxy groups and include glycidyl acrylate, glycidyl methacrylate, and allyl glycidyl ethersevet entre y samples

Examples of ethylenically unsaturated monomers that do not contain epoxy groups are alkyl esters of acerylloand methacrylicacid containing fro 101 to 20 atoms in the alkyl group. Specific examples of these acrylates 🖟 and methaciylates are methyl methacrylate; ethyl methacrylate; butyl methacrylate, ethyl acrylate, butyl acrylate, and 2-ethylhexyl acrylate. DITE OF WILDIES.

Examples of other copolymerizable ethylenically unsaturated monomers are as: described above for use with the tertiary amino-functional acrylic polymer, except that acid group-containing copolymerizable ethylenand methacrylic acid are preferably not used because of the possible reactivity of the epoxy and acid group. om length in twinted

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The advepoxide preferably has a number average molecular weight of from 376 to 3000, and more preferably of from 800 to 2000. This can be determined by the GRC method using a polystyrene standard. The polymer preferably has an equivalent weight per epoxy group (i.e., epoxy equivalent weight or EEW) of 188 to g 155.1500g and more preferably of 400 to ₹1000.991 (\$5.000 at the S. E. a fine reight). Rowner of user great, in this is

In order to form the microparticles according to the invention, the tertiary amino-functional acrylic polymer is first neutralized with an acid such as acetic acid or lactic acid.

After neutralization, the salted tertiary amino-functional acrylic polymer is blended with the polyepoxide. This blending can be carried out in the presence of polar organic solvent, a mixture of polar organic solvent the mand water, nonpolar organic solvent, or mixtures thereof. The blending can even be carried out in the absence of any solvent, such as in a mill, however, the blending is preferably carried out in the presence of polar organic solvent or a mixture of polar organic solvent and water (optionally with small amounts of nonpolar organic solwent). Examples of useful solvents for blending the components include butylcellosolve, ethyl cellosolve, and 550% etheros of glycols such as ethylene glycol, propylene glycol, or diethylene glycol, and mixtures thereof.

ம் அதன் After blending; the mixture of salted tirtiary amino-functional acrylic polymer, polyepoxidi, and any blendbabling solvent is dispersed in an aqueous medium to form an aqueous dispersion having particle siz s ranging ்ர if rom 0381/to 40 µm (pref rably 0.1 to 0,5 µm); and a nonwolatile content of from 10 to 40% by weight (preferably

20 to 30 % by weight). The aqueous medium will contain mainly water, but it may be desirable to add additional polar organic solvent, pH modifiers, surfactants, or dispersants to aid in formation of a dispersion having the desired particle size and uniformity. The us—of such solvents, pH modifiers, surfactants, and/or dispersants to form aqueous dispersions is well-known in the art, and does not require a detailed discussion herein.

The above particle size rang is represent preferred ranges for the dispersion of blended polyepoxide and quaternized acrylic as well as for the polymer microparticles. However, it is contemplated that significantly larger particlessizes at the blending stage may also be useful. The reason for this is that during the subsequent crosslinking step, the polyepoxide, which is substantially non-water dispersible; becomes part of a highly-charged water-dispersible crosslink matrix with the quaternized acrylic. Thus, significant reductions in particle size may be obtained during the crosslinking step.

In order to crosslink the blended material contained in each particle of the dispersion, the dispersion is then heated to a temperature of 60 to 98°C (preferably 78 to 82°C) for a time sufficient to crosslink the acrylic and the polyepoxide. This crosslinking occurs when the salted tertiary amine and epoxide react and quaternary groups are formed additional and the salted tertiary amine and epoxide react and quaternary groups are formed.

The resulting microparticle dispersion can then be incorporated into an electrodeposition coating bath. The microparticles are useful in the bath at levels of 4 to 20%, and preferably 3 to 10%, as a weight percentage of the principal resin nonvolatiles in the electrocoat bath. It is the principal resin nonvolatiles in the electrocoat bath.

The present invention is useful in cathodic electrodeposition coating compositions. Water-dispersible results in susable in the electrodeposition coating process may be classified, depending upon their dispersed state, into the solution type; the dispersion type; the emulsion type, and the suspension type. These types of resins are collectively referred to as "water-dispersible resins" herein. A wide variety of such resins are known and may be used in this invention. Include the state of the st

A variety of such resins are known including acrylic, polyester, polyether, phenolic, epoxy, polyurethane, polyamide, polybutadiene, and oil based resins. Typical examples thereof are acrylic copolymers containing acrylic or methacrylic acid, maleinized natural and synthetic drying oils, maleinized polybutadiene, half esters and half amides of maleinized oils and polymers. In the polymers are according to the polymers of the polymers of the polymers of the polymers.

Water-dispersible resins used in the cathodic electrodeposition coating process have a cationic functional group such as primary; secondary or tertiary amine moiety as a positively chargeable hydrophilic group. A variety of such resins are known including epoxy; polyether, polyester, polyurethane, polyamide, polybutadiene, phenolic and acrylic resins.

Cationic resins have been described in great number in the literature. They typically contain a number of basic groups, such as primary, secondary or tertiary amino groups, so as to provide dispersibility with water. If these resins contain primary and/or secondary amine groups, then they may not also contain hydroxyl groups and preferably they do. If only tertiary amino groups are present in the cationic resin, then the resin must contain hydroxyl or other functional groups in order to enable cross-linking. The amino equivalent weight of the cationic resin can range from 150 to 3000; and preferably 500 to 2000. The hydroxyl equivalent weight of the resins, if they have OH groups, is generally between 150 and 1000; and preferably 200 to 500. In addition, the resins may contain C=C double bonds, the C=C equivalent weight preferably being 500 to 1500.

FThe molecular weight (mean weight) of a typical cationic resin is usually in the range from 300 to:50,000, and preferably 5000 to 20,000. See that the condition of the Augustian Conditions of the Augustian Conditions of the Augustian Conditions of the Augustian Conditions of the C

Examples of cationic resins are described in the Journal of Coatings Technology, Vol. 54, No. 686, (1982), p. 33-41 ("Polymer Compositions for Cationic Electrodepositable Coatings"), the disclosure of which is incorporated by reference. Polymers of alpha, beta-olefinically unsaturated monomers that contain hydroxyl and/or amino groups may be introduced using appropriate monomers in the copolymerization, for example by means of hydroxyl or amino esters of alpha, beta-olefinically unsaturated carboxylic acids, such as hydroxyalkyl (meth)-acrylates or aminoalkyl (meth)acrylates, or by polymeranalogous reaction with diamines or polyamines; for example with N,N-dimethylaminopropylamine, with formation of amide, amino or urethane groups. The polyaminopolyamides, which can be obtained from dimerized fatty acids and polyamines, are a further group. Aminopolyether polyols, which can be prepared by reaction of primary or secondary amines with a polyglycidyl ether, are particularly suited for this. Sufficient epoxide groups to convert all amino groups into tertiary amino groups are advantageously present here. The preferred polyglycidyl ethers are polyglycidyl thers of bisphenot A and similar polyphenols. They can by prepared, for example by etherifying a polyphenol using an epihalohydrin, such as epichlorohydrin, in the presence of alkali.

The polyglycidyl ethers of the polyphenols may be reacted as such with the amines; but it is frequently advantageous to react some of the reactive epoxide groups with a modified material in ord rate improve the film properties. The reaction of the epoxide groups with a polyol or a polycarboxylic acid is particularly preferred.

Useful polyols can include polyether polyols, polyester polyols, or urethane polyols. Polyether polyols can

be prepared by addition polymerization of alkylene oxides (for example ethylene oxide, propylene oxide, tetrahydrofuran) with low-molecular-weight polyols having 2 to 8 carbon atoms and a molecular weight of about 50 to 300 (f r example ethyl ne glycol, diethylene glycol; propylene glycol, dipr pylene glycols, glycerol, trimethylolpropan , 1,2,6-hexanetriol, pentaerythrit). If ethylene oxide is used alone or in combination with other 5 44 alkylene oxides as alkylene oxide components; the water-solubility of the resin is improved. Polyester polyols can be prepared by reaction of the above mentioned low-molecular weight polyols or epoxy compounds, for example fatty acid glycidyl esters, with polycarboxylic acid (for example adipic acid, succinic acid/maleic acid/phthalic acid/porterephthalic acid)/or derivatives thereoformal deda printeress. Polyester polyols can be prepared by ring-opening polymerization of a cyclic ester such as caprolactone or but vrolactone. the gradient sent and girlur barrando in a Urethane-modified polyls can be obtained by reaction of an excess of the abovementioned polyether polyols of polyester polyols with an organic polyisocyanate along 1. இரு நின்ற வழின் முறைய மார் நான்ற நின்ற வழிகள் The above-mentioned polycarboxylic acids are obtained by reaction of the polycle described above with an excess of polycarboxylic acids or, preferably, the anhydrides thereof. They can likewise be obtained by esterification of polycarboxylic acids, or antiverides thereof, using low-molecular weight polycle, such as ethylene eglycol; propylene glycol; etc. Low-molecular weight polyether polyamines or polyamines; such as; for example, hexamethylenediamine, may also be employed in place of the low-molecular weight polyols.is (so the were this The modification of the aminopolyether polyets using polyets or polyearboxylic acids is preferably carried efcrout before the reaction of the polyglycidy ethers with the primary or secondary amaines. However, it is also 20 possible to select the ratio of the polyglycidy bether used as starting material to the amines in such a fashion That an excess of epoxy groups is present. The epoxy groups may then be reacted with the polycarboxylic acids or polyols. It is furthermore possible to further modify the final product, which no longer contains epoxid selection of the hydroxyl groups with glycidyl ethers in award one suiter nous to vience A performance of the curing mechanism of particular resins, they may be classified into three classes. The first 25% Forter is those capable of self-crosslinking through a radical or exidative polymerization reaction. The second class of resins requires a crosslinking agent such as blocked polyisocyanates. The third one utilizes both the ि अ**self-crosslinking reaction and the crosslinking agent in odmbination**t में beau snice melder equipment कि स्र A coording to the type of energy source required for initiating the crosslinking reaction, the water-dispersible chargeable resins may also be classified into the ambient temperature curing or more preferably heatcuring. armer on, soe fine ador. The water-dispersible resins useful as principal resins in the present invention are typically hydrophilic s such that they are not soluble or dispersible in water when they are in the form of a free base; but become soluble or dispersible to make a stable aqueous solution or dispersion when a sufficient amount (e.g., at least 320%; and more typically:50%) of the base function is neutralized. If the water-dispersible resins are too hy-35 gedrophilic/they/fail-to-form/a coating film having satisfactory water-to-corrosion resistance and/or the applicaingletion of electrodeposition coeffing processes becomes difficultar 071 most spars and sear of resista entita h ofder to enhance various film properties, the water-dispersible resins are often used in the form of an emulsion in which the water-dispersible resinconstitutes a continuous phase, and an optional water-insoluble constitutes a dispersed phase, resin free from constitutes a dispersed phase. When the resin can be crosslinked with a crosslinking agent included in the coating composition for the 1509 electrocoat primer layes, any of a number of crosslinking agents on curing agents may be used. Commonlyworksed crosslinking agents include blocked polyisocyanates including isocyanaurates of polyisocyanates (e.g., and transesterification crosslinking agents. A 1971 4 Annable of the appropriate stable outre a prefetred embodiment of the invention, the crosslinking agent is an aromatic polyisocyanate, including 45/65 isocyanurates of aromatic polyisocyanates. Useful aromatic polyisocyanates include toluene diisocyanate (TDI), methylene diphenyl diisocyanate (MDI), tetramethylxylene diisocyanate, and the like. In another preferred embodiment, an isocyanurate of an aliphatic polyisocyanate such as hexamethylene diisocyanate is used. These isocyanates are pre-reacted with a blocking agent such an oxime, an alcohol, or an amine, which blocks The isocyanate crosslinking functionality, Upon heating, the blocking agents separate and crosslinking occurs. 56.000 is The electrodepositable coating compositions of the present invention are dispersed in aqueous medium. The term "dispersion" as used within the context of the present invention is believed to be a two-phase translucent or opaque aqueous resinous system in which the resin is in the dispersed phase and water the continuous phase. The average particle size diameter of the resinous phase is about 0.1 to 10 microns, preferably enseless than 5 microns: The concentration of the resinous products in the aqueous medium is, in general, not crit-55/: e ical, but of dinarily the major portion of the aqueous dispersion is water. The aqueous dispersion usually conanisatains from about 3 to 50 percent preferably 5 to:40 percent by weight resin solids. Aqueous resin concentrates which are to be further dilut d with water, generally range from 10 to 30 percent by total weight solids.

anodic electrodeposition or an acid in case of the cathodic lectrodeposition in an amount sufficient to neutralize enough of the ionic groups to impart water-dispersibility to the resin. Examples of bases include ammonia, diethanolamine, triethanolamine, methylethanolamine, diethylamine, morpholine, and potassium hydroxide. Examples of acids include phosphoric acid, acetic acid, propionic acid and lactic acid.

Besides water, the aqueous medium may also contain a coalescing solvent. Useful coalescing solvents include hydrocarbons, alcohols, esters, ethers and ketones. The preferred coalescing solvents include alcohols, polyols and ketones. Specific coalescing solvents include monobutyl and monohexyl ethers of ethylene glycol, and phenyl ether of propylene, glycolethylcellosolve, propylcellosolve, butylcellosolve, ethyleneglycol dimethyl ether, or diacetone alcohol. A small amount of a water-immiscible organic solvent such as xylene, toluene, methyl isobutyl ketone or 2-ethylhexanol may be added to the mixture of water and the water-miscible organic solvent. The amount of coalescing solvent is not unduly critical and is generally between about 0 to 15 percent by weight, preferably about 0.5 to 5 percent by weight based on total weight of the resin solids.

The electrodeposition coating composition used in this invention may further contain conventional pigments such as titanium dioxide, ferric oxide, carbon black, aluminum silicate, precipitated barium sulfate, aluminum phosphomolybdate, strontium chromate, basic lead silicate or lead chromate. The pigment-to-resin weight ratio can be important and should be preferably less than 50:100, more preferably less than 40:100, and usually about 20 to 40:100. Higher pigment-to-resin solids weight ratios have also been found to adversely affect coalescence and flow.

The electrodeposition coating compositions used in the invention can contain optional ingredients such as wetting agents, surfactants; UV absorbers; HALS compounds, antioxidants; defoamers and so forth. Examples of surfactants and wetting agents include alkyl imidazolines such as those available from Ciba-Geigy Industrial Chemicals as Amine C®, acetylenic alcohols available from Air Products and Chemicals as Surfynol® 104. These optional ingredients, when present, constitute from about 0 to 20 percent by weight of resin solids. Plasticizers are optional ingredients because they promote flow. Examples are high boiling water immiscible materials such as ethylene or propylene oxide adducts of nonyl phenols or bisphenol A. Plasticizers can be used and if so are usually used at levels of about 0 to 15 percent by weight resin solids.

Curing catalysts such as tin catalysts are usually present in the coating composition. Examples are dibutyltin diffaurate and dibutyltin loxide: When used, they are typically present in amounts of about 0.05 to 2 percent by weight tin based on weight of total resin solids.

In general, sufficient water is added so that the dispersion has a solids content of more than 20; preferably more than 30% by weight.

The electrodeposition coating composition used in this invention may be applied on a conductive substrate by the electrodeposition coating process at a nonvolatile content of 10 to 25% by weight to a dry film thickness of 10 to 35 µm. After application, the coating may be oured at ambient or an elevated temperature, depending upon the nature of particular base resins.

The electrodeposition of the coating preparations according to the invention may be carried out by any of a number of processes known to those skilled in the art. The deposition may be carried out on all electrically conducting substrates, for example metal, such as steel, copper aluminum and the like.

According to the invention, a pigmented resin coating and optionally a clearcoat layer is applied over the electrocoat primer layer. In automotive applications, the pigmented resin layer is often called a basecoat or pigmented basecoat. The resin in the pigmented resin layer can be of a number of resins known in the art. For example, the resin can be an acrylic, a polyurethane, or a polyester. Typical pigmented resin coating formulations are described in U.S. Patents 4,791,168, 4,414,357, and 4,546,046. The pigmented resin can be cured by any of the known mechanisms and curing agents, such as a melanting polyol reaction (e.g., melanifee cure of a hydroxy-functional acrylic resin).

The invention is further described in the following examples.

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Examples - Electrocoat Coating Compositions and the contract of the contract o

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To a 5 liter round bottom flask equipped with a condenser, Nitrogen flow, and temperature probe, the following materials were added:

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997.5 g 2,-4 toluen di-isocyanate (Mondur TD-80®)

To an addition tank the following was added:

828.3 g h xyl cellosolve

The hexyl cellosolv was added to the flask at a rate in order to maintain an exotherm temperature less than 50°C. The temperature was maintained at 45°C for an additional hour at which time the following was added:

	0.5 g நில rdibutyl tin dilaurateh முழுமை அடி நிக்கப்பட்ட செலகம் நில மக்க கொண்ணைக்கு செல் நி
• •	The following was added at a rate that caused the temperature to ris to and maintain at 115 - 120° C.
	256.6:g. instrimethyol propane southers are a function for the precious contract the engage source of the contract of the cont
	The mixture was maintained at 120°C for an additional two hours. The mixture was cooled to 110°C at which
5 ≒	time the following was added with continued mixing to cool the resin: (a) And (b) And (b) And (b) And (c) And
	826.1 g Transmethylrisobutyl ketone a fact is about the control of
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:	To a 5 liter round bottom flask equipped with a condenser, Nitrogen flow, and temperature probe, the following
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	The material from the addition tank was added to the flask over two hours. The temperature climbed to 60°C
$f^{\dagger}N$	and was maintained during the first hour. The temperature increased to 118°C by the end of the second hour.
20 :::	The batch was maintained at \$18% for three hours after which the following was added to a 1870 in three hours after which the following was added to a 1870 in three hours after which the following was added to a 1870 in three hours after which the following was added to a 1870 in three hours after which the following was added to a 1870 in three hours after which the following was added to a 1870 in three hours after which the following was added to a 1870 in three hours after which the following was added to a 1870 in three hours after which the following was added to a 1870 in three hours after which the following was added to a 1870 in three hours after which the following was added to a 1870 in three hours after which the following was added to a 1870 in three hours after which the following was added to a 1870 in three hours after which the following was added to a 1870 in three hours after which the following was added to a 1870 in three hours after which the following was added to a 1870 in three hours after which the following was added to a 1870 in three hours after which the following was added to a 1870 in three hours after which the following was added to 1870 in three hours after which the following was added to 1870 in the 1870 in three hours after which the following was added to 1870 in three hours after which the 1870 in th
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-33	and the consistence as the second state are usually prepared in the continuon municipal examination with
H^{μ} a	To a 12 liter round bottom flask equipped with a condenser, nitrogen flow, and temperature probe, the following
	materials were added: souther maintain a library to be as a place of the materials were added.
30 ψ^{\prime}	963:8 95 nan DGEBA main a cultura sast popular, year that the proposition of the proposition 200 863:8
	348.2 g ethoxylate of bisphenol A
4.35	17312 graftigh xylenes besking in digeminatinen. Ferdinatio op bis ienn avsicht sociativa eposition terrapione
	The mixture was agitated and heated to 120°C and vacuum distilled into a Dean Stark trap to remove any water.
4. 40	The following was:added and the mixture was heated to 135° Case 36 to most lique latter in 28 to 47 to
35	280.1 g bisphenol A September 9 sind askinding for a vice on the control of the c
14 g	/2.2igaco cem benzyl:dimethyl:amine in ohibrooce கூரார் உள்ளை உள்ள விழுக்கும் கண்குக்கு ஆட்கும். ஓ
$\chi^{\alpha\beta\gamma\gamma}$	The mixture exothermed to 172°G and was cooled to and maintained at 143°C for two hours from the point of
	peak temperature. The following was added on feet and the first authorization and the common was
200	2.6 grand benzyl dimethyl amine in toluta ons grant in the creater, and bit over entert order access.
40	sine epoxy concentration was titrated at thirty minute intervals to an endpoint of 1200,g. N.W. resin/eq. epoxide
10月	at which point the following was added to the separate to the study of the state of the constraint of the separate to the separate of the sepa
وتر أبت	e 1986.5 g udo re component #1 a la fill reservator de culturar altre consilie de la sucitada de major desendada e consilie de la consilie del la consilie de la consilie
138 Yu	#At 105°C, the following: was added in the order listed: ↓ ↓ □ → □ ≥ □ □ □ □ □ □ □ □ □ □ □ □ □ □ □ □
50.54.1	107.0 g. a ketimine of diethylene triamine (70% solution) promotion promotion as a second control of the control of t
45	4.6 g phenyl ether of propylene glycol
	74.0 g methyl ethanol amine: அதிர் முக்கி நடிகள் இருந்தி நடிகள் நடிகள் நடிகள்
	The mixture exothermed to 118°C and was maintained at that temperature for one hour. The resin was cooled
	to 90°C with the addition of the following:
	293.7 g component #2
50	Once homogeneous, the following was added in the order listed with increased mixing:
	98,0 g lactic acid 85%
117 •	40.6 g: ுsurfynol/104 (50% solution)க்கும் பிறு செத்த நடிக்க நடிக்க கூடுக்க கடிக்க
	Once homogeneous, the following was added over 15 minutes:
	3113.1 g deionized water
55	Once homogene us, the following was added:
	408.0 g deionized wat r
13.11	Once-homogeneous, the following was added to what are also has been a product the contract of
1929	266,0 gp-two-deionized waterwise rubit some flobe as not Drubbye (see that the contract of the

	Once homogeneous, the following was added: 266.0 g deionized water	figures as a sign	
	Once homogeneous, the following was added: heavily the Ale		
	266.0 g deionized water		
5	•	· · · · · · · · · · · · · · · · · · ·	
9	Th mulsion was stirred in an open container for 3.5 weeks	to allow evaporation of low boiling solvents. Ev	ар-
	oration loss was replenished daily with distilled water during		
	Description of the state of the	京在《花》《 新闻 诗》(1) (1) (1) (1)	
	Preparation for component 4:	·	
		1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1	
10	To a 5 liter round bottom flask equipped with a condenser, ni	trogen flow, and temperature probe, the follow	ing
		State San Comment	
	1606.0 g 2,4-toluene di-isocyanate	the Space of the great	
	129.2 g methyl isobutyl ketone	5 TE 10 10 10 10 10 10 10 10 10 10 10 10 10	
	To an addition tank the following was added:	THE MORE STORE	
15 -	் 1231.9 ga ik. i2-ethyl hexanoli கம்சம் ilsa c s no griffsa ye s	anolidi tudin e ji sici babuni asim indigisirbi di j	
	The alcohol was added to the flask at a rate such that the t	temperature was maintained between 40 - 43	°C
	Once the addition was complete the temperature was maintain	ned at 43°C for two hours: after which the follow	ina
	was added:		9
. •	"32.3·g" simethyldscoutylaketone≪ That gravia aseneurocke.	third require designments that a series a	
20		matering werelt datids:	
	Preparation for component 5:	1415 Ale Methy ene dispryange	
		the diluerate	
	To a 5 liter round bottom flask equipped with a condenser, Ni		: !
	materials were added:	nogenhow, and temperature probe, me foliowing the land land to a classical control of the contro	ing
25		or the loop and the control of the	
	The following was added over 4.5 hours: 0.08 - 13 to Coules	State of the section	
	1463.9 g component #4		
	•	only which said in the sea for the way exact first and	
	The temperature rose to and was maintained between 73 -	76°C during this addition. The temperature w	as
30	maintained an additional 30 minutes at 77°C at which time the		
30	178.8 g butyl cellosolve	Tree flooring was priced or and tree stand	1/
	After an additional 20 minutes the following was added:	acomo សក្សាមែលមិន ក្រុងការ។	
	536.3 g (*** Clacific acid (85%)) (***) Is the content of the second of		
	423.1 g a file delonized water a box as have this state and a	CONTRACTOR OF THE STATE OF THE	
	The mixture was maintained 2.5 hours at 91°C.		
35			7.
	Preparation for component 6:	- Jacobs Frysia, et watera il pieddit	
	To a 5 liter round bottom flask equipped with a condenser, nit	rogen flow, and temperature probe, the following	ng
	materials were added:		3
10	″639.3 g 27 95 DGEBA¢#™63.6 .woll @cc a i i # ar jun6, a c	Balle nuc bely sine on serve	با
-	260.7 g bisphenol A	de nos erew gisheter.	
	The batch was heated to 110°C at which time the following was	vas-addedy its end showcost to be table	
		Conf. 4 of the record year one or	
14.	-1:0 g and xylene to any way has at the totally same or anno		
15	The mixture exothermed to and was maintained at 180°C for		nA
	was added after which the temperature was maintained for o	one hour.	ııy
	0.2 g triphenyl phosphine a 4 16 to the visite 4 minutes and		
	1.0 g xylene	would begree get	
	The batch was cooled to 132°C at which time the following w		
i0	371.4 grade component #4888 grade section and an order of		
-	3.2 g xyl ne		13
		2 7 JOSEP 4	
	The batch was maintained at 124°C for two hours after which		
	1070.0 g butyl cellosolve	वीर्व १७ व व्यक्ति को तथा जाता नहीं	
_	The mixture was cooled to 82°C at which time the following v		
5	517.2 g 11.15 Component #5. A 7 1 9888 978 1 A 2 2 4 9 8 5		ΑĊ.
	136.0 g butyl cellosolve	व उत्पादक स्थान स्थानिक विद्यालय	
	The mixture was maintained for five hours at 82°C to comple		
		A heredyská – grá 946	

EP 0.612.818.A

In a staint is steel 1/2 gallon vess i, the following were added, and mixed with a high ap, ed-cowless blade for 15 minutes: 5 923.0 g			
In a stain! as steel 1/2 gallon vess I, the following were added and mixed with a high ap ed cowles blade for 15 minutes: 5 23.0 g		Preparation for component 7:	to the second second second properties of
In a stain! as steel 1/2 gallon vess I, the following were added, and mixed with a high sp. ed. cowless blade for 15 minutes: 5 623.0 g			•
15 minutes: 5 923.0 gdelonized water 374.8 g component #6 7.7 g anti-crater additive After a homogeneous sate was obtained, the following components were added in the order-fisiteds: 2.5.3 g carbon black 60.7 g lead silicate 18.1 g day extender 838.6 g TiO2 117.8 g delonized water 19.1 The material was mixed for one hour followed by milling on a small media milit to a flyaness of grind of 11 µm. The material was mixed for one hour followed by milling on a small media milit to a flyaness of grind of 11 µm. The material was mixed for one hour followed by milling on a small media milit to a flyaness of grind of 11 µm. To a 5 liter round bottom flask equipped with a condenser, nitrogen flow, and temperature probe, the following materials were added: 1142.5 g Methylene dilsocyanate 2.0 g dibutyl tin diluarate 1142.5 g diethylene glycol butyl ether 252.5.5 g diethylene glycol butyl ether 253.5 g diethylene glycol butyl ether 254.5 g amethylisobutyl ketone 255.5 g amethylisobutyl ketone 256.5 g amethylisobutyl ketone 257.5 g amethylisobutyl ketone 258.5 g amethylisobutyl ketone 259.6 g trimethylol propane 259.6 g trimethylol propane 259.7 (or 4 additional hours. The following was added afterwhich the temperature was maintained at 8°C to 4 additional hours. The following was added afterwhich the temperature was maintained at 8°C to 4 additional hours. The following was added afterwhich the temperature was maintained at 8°C to 4 additional hours. The following was added afterwhich the temperature was maintained at 8°C to 4 additional hours. The following was added afterwhich the temperature was maintained at 8°C to 4 additional hours. The following was added afterwhich the temperature was maintained at 8°C to 4 additional hours. The following was added afterwhich the temperat		In a staint ss steel 1/2 gallon vess 1, the following were	acided and mixed with a high on advanded blade for
5 22.0 g component #6 7.7 g anti-crater additive After a homogeneous sate was obtained, the following components were added in the order fisted: 25.3 g carbon black 314.9 day extender 39.8 g TiO2 117.6 g desimized water 15 The material was mixed for one hour followed by milling on a small media mill to a figures or grid of £1 µm. Preparation for component 80 10 a 5 liter round bottom flask equipped with a condenser, nitrogen flow, and temperature probe, the following: 16 a 5 liter round bottom flask extractions at the following was added dover Alway beyond the mixture was diluted with the following: 17 a 5 liter round bottom flask equipped with a condenser, nitrogen flow, and temperature probe, the following: 18 a 337.4 g ethylene discopanate discopanate and the following was added dover Alway bottom flask equipped with a condenser of the following was added dover Alway bottom flask equipped with a condenser of the following was added dover Alway bottom flask equipped with a condenser of the following was added dover Alway bottom flask equipped with a condenser of the following was added and the addition. 18 a first was flasted to 4982 at which time the following was added dover Alway bottom flask equipped with a condenser of the following was added at a first flowing was added over 30 minutes: 19 a first was flasted as a first flowing was added at flast which the lemperature was maintained at 87°C for 4 additional hours. The following was added after which the lemperature was maintained at 87°C for 4 additional hours. The following was added after which the lemperature was maintained at 87°C for 4 additional hours. The following was added after which the lemperature was maintained at 87°C for 4 additional hours. The following was added after which the lemperature was maintained at 87°C for 4 additional hours. The following was added after which the lemperature was maintained at 87°C for 4 additional hours. The following was added after which the lemperature was maintained at 87°C for 4 additional hours. The fo	•	15 minutes:	
374.6 g component #6 7.7 g anti-crater additive After a homogeneous sate was obtained, the following components were added in the order-fisted: 25.3 g carbon black 10. 43.4 g: dibuty thin oxide. 10. 19. 43.4 g: dibuty thin oxide. 11. 6 g designized water 11. 6 g designized water 11. 7 g designized water 12. 11. 8 g designized water 13. 11. 8 g designized water 14. 12. 8 g designized water 15. The material was mixed for one hour followed by milling on a small model and it to a finances of grind of 11 µm. 15. The mixed was mixed for one hour followed by milling on a small model and it to a finances of grind of 11 µm. 16. The mixed was mixed for one hour followed by milling on a small model and it to a finances of grind of 11 µm. 17. The mixed was mixed for one hour followed by milling on a small model and the addition of the washed was added. 18. 19 g dibuty thin dilusarate 19. The mixed revas-heated to 490, at which time the following was added, over-two-bours one, the following was added, over-two-bours one, the following was added over-two-bours one, the following was added over-two-bours one transported by the following was added from an addition tank a	5		- 17 - 17 - 17 - 17 - 17 - 17 - 17 - 17
After a homogeneous sate was obtained, the following components were added in the order listed: 25.3 g carbon black 43.4 g dibuty the oxide a warring of the sate of the sat	•	374.6 a component #6	等。 \$P\$ \$P\$ 14.4 (1995年) 1995年, 2006年, 2006年, 2006年, 2006年,
After a homogeneous sate was obtained, the following components were added in the order/fisted: 25.3 g carbon black 39.4 g dibutyf tin oxide: 83.9 c TiO2 117.6 g deionized water The material was mixed for one hour followed by milling on a small media mit to affixeness of grand of 1 µm. 15 The material was mixed for one hour followed by milling on a small media mit to affixeness of grand of 1 µm. 16 Preparation for component 8: 17 a 3 Filter round bottom flask equipped with a condenser, nitrogen flow, and temperature was maintained at 87°C for 4 additional hours. The following was added over 30 minutes: 18 377.4 g thylene glycol butyl ether 20 g dibutyl tin dilutarate 21 a dibutyl ting dilutarate was maintained at 57 - 60°C until one hour, following the end of the addition. The mixture was diluted with the following: 18 26.8 g methyl isobutyl ketones Preparation for component 8: 19 37.4 g thylene glycol butyl ether 21 g dibutyl ting dilutarate was maintained at 57 - 60°C until one hour, following the end of the addition. The mixture was diluted with the following: 18 26.9 methyl isobutyl ketones Preparation for component 8: 19 30 The following was added over 30 minutes: 19 4.6 g trimethylol propane 19 4.7 a 3 liter round bottom flask equipped with a condenser, nitrogen flow, and temperature probe, the following materials were added: 19 4.7 g methyl isobutyl ketone 19 4.8 g methyl isobutyl ketone 19 4.9 g incovanurate of HMDhs@esmodur N3300®) 20 367.4 g methyl isobutyl ketone 21 g dibutyl tin dilutarate 22 a dibutyl tin dilutarate 23 68 g n-butanol 24 12 flee round bottom flask equipped with a condenser, nitrogen flow, and temperature probe, the following materials were added: 25 67.5 g dibutyl tin dilutarate 26 7 a 3 liter round bottom flask equipped with a condenser, nitrogen flow, and temperature was maintained at 80°C for 1 hour at which time no free isocyanate was observed by infire-red spectrescopy. 27 Prefaration for component 10: 28 7 a 12 flee round bottom flask equipped w		77a onti croter additivo	ाक्षात्राहरीत पुरस्कान्त्रीहरू ता नाम्ब्रम कर्मुका शरू से अस्तर नहीं तात्रात्र
25.3 g dishtyt in dislarate: which time the following was added over 30 bithyt fin dislarate: which is following was added over 30 minutes: 337.6 g trimethylol propane TREDION was added over 30 minutes: 34.6 g trimethylol propane The following was added over 30 minutes: 34.6 g trimethylol propane The following was added over 30 minutes: 34.6 g trimethylol propane The preparation for component 8: 337.4 g methyl isobutyl ketone The printiture was allowed to exotherm to and was maintained at 77°C during this addition. The temperature was maintained at 8°C. 38.3 g trimethylol propane The following was added over 30 minutes: 34.6 g trimethylol propane The preparation for component 8: 337.4 g methyl isobutyl ketone The temperature was allowed to exotherm to and was maintained at 77°C during this addition. The temperature was maintained at 8°C. 38.3 g to a bithylol propane The temperature was allowed to exotherm to and was maintained at 77°C during this addition. The temperature was maintained at 8°C. 38.3 g to a bithylol propane The temperature was allowed to exotherm to and was maintained at 77°C during this addition. The temperature was maintained at 8°C. 38.3 g to a bithylol propane The temperature was allowed to exotherm to and was maintained at 77°C during this addition. The temperature was maintained at 8°C. 38.3 g to a bithylol propane The temperature was allowed to exotherm to and was maintained at 77°C during this addition. The temperature was maintained at 8°C. 38.3 g to a bithylol propane The temperature was allowed to exotherm to and was maintained at 77°C during this addition. The temperature was maintained at 8°C. 38.3 g to a bithylol propane The temperature was allowed to exotherm to and was maintained at 8°C. 38.3 g to a bithylol propane The temperature was allowed to exotherm to and was maintained at 7°C during this addition. The temperature was maintained at 8°C. 38.3 g to buthylol propane The temperature was maintained at 8°C. 38.3 g to buthylol propane The temperature wa			
43.4.9g deal silicate 18.19 lead silicate 18.19 clay estender 83.9.8 g TiO2 117.6 g deionized water 117.6 g deionized water 117.6 g deionized water 118.10 clay estender 118.10 c		After a nomogeneous sate was obtained, the following	components were added in the order listed:
50.7 g lead silicate 18.1 g day extender 839.6 g TiO2 117.6 g deionized water 15 The material was mixed for one hour followed by milling on a small media millit configures of gird of 11 µm. Preparation for component 8: To a 5 liter round bottom flask equipped with a condenser, nitrogen flow, and temperature probe, the following materials were added: 142.5 g Methylene discovanate 2.0 g dibuty thin diluarate 2.1 Jhe mixture was allowed to 40.90, at which time the following was added over 3 minutes: 2.5 g dethylene glycol propyle ther The temperature increased to and was maintained at 57 - 60°C until one hour following was added over 30 minutes: 2.1 g dibuty this diluarate: 3.0 The following was added over 30 minutes: 3.1 The following was added over 30 minutes: 3.2 The following was added over 30 minutes: 3.3 The following was added over 30 minutes: 3.4 For 4 additional hours. The following was added a free which the temperature was maintained at 8°C, for 4 additional hours. The following was added a free which the temperature was maintained at 8°C, and a supplied over 30 minutes: 3.3 Between the following was added from an addition and was maintained at 7°C for 4 additional hours. The following was added a free which the temperature was maintained at 8°C, and a supplied with a condenser, nitrogen flow, and temperature was maintained at 8°C for 4 additional hours. The following was added a free which the temperature was maintained at 8°C for 4 additional hours. The following was added free which the temperature was maintained at 8°C for 4 additional hours. The following was added free which the temperature was maintained at 8°C for 4 additional hours. The following was added free which the temperature was maintained at 8°C for 4 additional hours. The following was added free which the temperature was maintained at 8°C for 4 additional hours. The following was added free which the temperature was maintained at 8°C for 4 additional hours. The following was added free which the temperature was maintained at 8°C fo			
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176 g delonized water 176 The material was mixed for one hour followed by milling on a small media mill to affiganess of gindr of 11 μm. 176 Preparation for component 8: 170 a 5 liter round bottom flask equipped with a condenser, nitrogen flow, and temperature probe, the following materials were added: 170 a 5 liter round bottom flask equipped with a condenser, nitrogen flow, and temperature probe, the following materials were added: 170 a 5 liter round bottom flask equipped with a condenser, nitrogen flow, and temperature probe, the following materials were added: 1712.5 g Methylene disocyanate disocyanate disorders were supported by the disorders were s		• • • • • • • • • • • • • • • • • • • •	and the second of the second o
117.6 g deionized water The material was mixed for one hour followed by milling on a small media mill to a figuress of grind of 11 µm. Preparation for component 8: To a 5 liter round bottom flask equipped with a condenser, nitrogen flow, and temperature probe, the following materials were added: To a 5 liter round bottom flask equipped with a condenser, nitrogen flow, and temperature probe, the following materials were added: The smixture transheated to 40°C attainment the following was added given two hours as a smith probability of the structure in the temperature in the temperature was maintained at 57 - 60°C until one hour following the end of the addition. The mixture was diluted with the following: The temperature increased to and was maintained at 57 - 60°C until one hour, following the end of the addition. The mixture was diluted with the following: The following was added over 30 minutes: 94.6 g trimethylol propane The temperature was allowed to exotherm to and was maintained at 77°C during his addition. The temperature was maintained at 87°C for 4 additional hours. The following was added after, which the temperature was maintained at 87°C for 4 additional hours. The following was added after, which the temperature was maintained at 87°C for 4 additional hours. The following was added after, which the temperature was maintained at 87°C for 4 additional hours. The following was added after, which the temperature was maintained at 87°C for 4 additional hours. The following was added from an addition tank at a rate such that the temperature probe, the following materials were added: Preparation for component 9: **The temperature was maintained for 30 minutes after which the temperature probe, the following materials were added: **The mixture was heated to 75°C for 1 hour at which time no free isocyanate was observed by infra-red spectroscopy. Pregignation for component 10: **The mixture was heated to 75°C for 1 hour at which time no free isocyanate was observed by infra-red spectroscopy.		•	manyodas basawari si in tolkeri
The material was mixed for one hour followed by milling on a small media mill to adjuve and before the component 8 and the analysis are understanded and the component 8 and the component			subject to stock to the service of their
The material was mixed for one hour followed by milling on a small media mill to authorize and another milling and a small media milling and a small media milling authorize and milling. Preparation for component 8: "The mixture was determined and the safetime and milling authorize and mixture was maintained at 57 and 5 liter round bottom flask equipped with a condenser, nitrogen flow, and temperature probe, the following materials were added: 142.5 g Methylene diisocyanate 2.0 g dibutyl tin dilurarate 2.1 Jhe mixture was abstacled to 40°C, at which time the following was added over two-hours and a content of the addition. The mixture was diluted with the following: 25.5 g diethylene glycol propyl ether The temperature increased to and was maintained at 57 - 60°C uetil one hour following the end of the addition. The mixture was diluted with the following: 27.1 g dibutyl tin dilurarate; which the safe and of the addition. The mixture was diluted with the following: 27.1 g dibutyl tin dilurarate; which the safe and of the addition. The temperature was allowed to exotherm to and was maintained at 7°°C during this addition. The temperature was allowed to exotherm to and was maintained at 7°°C during this addition. The temperature was maintained at 8°°C for 4 additional hours. The following was added after which the temperature was maintained one hour at 85°C. 28.3 83.4 g n-butanol 30.0 g methyl isobutyl ketone 29.4 g isocyanurate of HMDIs(Desmodur N3300®) and temperature probe, the following materials were added: 20.4 g dibutyl amine 20.5 methyl isobutyl ketone 20.6 g n-butanol 20.6 g n-butanol 20.7 methyl isobutyl ketone 20.7 methyl isobutyl ketone 20.8 g n-butanol 20.9 dibutyl amine 20.9 dibutyl amine 20.0 dibutyl amine 2			heros sawlari valot sib žijes sodinte (1. n.)
Preparation for component 8: To a 5 liter round bottom flask equipped with a condenser, nitrogen flow, and temperature probe, the following materials were added: 1142.5 g Methylene diisocyanate dibuty tin diluarate dibuty tin diluarate dibuty tin diluarate crass-heated to 40°C attwhich-time the following was added over two-hours and cause and following the end of the addition. The mixture crass-heated to 40°C attwhich-time the following was added over two-hours and cause and following the end of the addition. The temperature increased to and was maintained at 57 - 60°C uetitl one hours following the end of the addition. The mixture was diluted with the following: 1782.6 g methyl isobutyl ketons of the end of the addition. The following was added over 30 minutes: 1782.6 g trimethylol propane the following was added after which the temperature was maintained at 87°C for 4 additional hours. The following was added after which the temperature was maintained at 87°C for 4 additional hours. The following was added after which the temperature was maintained one hour at 85°C. 1883.4 g n-butanol do the flask equipped with a condenser, nitrogen flow, and temperature probe, the following materials were added: 1897.4 g methyl isobutyl ketone the following was added after which the temperature was maintained at 80°C. 1897.4 g methyl isobutyl ketone the following was added from an addition tank at a rate such that the temperature probe, the following materials were added: 1897.5 g dibutyl amine the temperature was maintained to 30°C or 1 hour at which time no free isocyanate was observed by infra-red spectroscopy. 1898.6 g n-butanol flask equipped with a condenser, Nitrogen flow, and temperature probe, the following materials were added: 1898.6 g n-butanol flask equipped with a condenser, Nitrogen flow, and temperature probe, the following materials were added:		The material was mixed for one hour followed by milling	on a small media mill to a finaness of grind of 11 µm.
To a 5 liter round bottom flask equipped with a condenser, nitrogen flow, and temperature probe, the following materials were added: 1142.5 g	٠٠.	on that the temporrature was ordrigans in between NU (4)	the same a contest and of behing how tertains. In the
To a 5 liter round bottom flask equipped with a condenser, nitrogen flow, and temperature probe, the following materials were added: 1142.5 g Methylene diisocyanate 2.0 g dibutyl tin diluarate 2.1 p diethylene glycol butyl ether 525.5 g diethylene glycol butyl ether 1 the temperature increased to 40°C at which time the following was added over two hours and a substance of the properties	. 7,74	Preparation for component 8: 10 (2013) is pensioned as well	Stinsten on a training son manche advance
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2.0 g dibutyl tin diluarate In Jhe mixture twas heated to 40%C at which time the following was added over two-hours one control of the property of the proper		1142.5 g Methylene diisocyanate	A transported in the control of
Je mixture was heated to 40°C at which time the following was added over two-hours on the state of 525.5 g diethylene glycol butyl ether the beautiful of the temperature increased to and was maintained at 57 - 60°C until one hour following the end of the addition. The mixture was diluted with the following: 782.6-9 smethyl isobutyl ketons. The following as added over 30 minutes: 94.6 g trimethylol propane The temperature was allowed to exotherm to and was maintained at 7°C during this addition. The temperature was maintained at 8°C for 4 additional hours. The following was added after which the temperature was maintained one hour at 85°C. 83.4 g n-butanol 30.0 g methyl isobutyl ketone Preparation for component \$1 socyanurate of HMDH (Desmodur N3300®) 387.4 g methyl isobutyl ketone Preparation for component of the mixture was maintained one of the mixture was maintained at 7			CONTROL OF THE CONTRO
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Pregaration for component 10: To a 12 fiter round bottom flask equipp d with a condenser, Nitrogen flow, and temperature probe, the following materials were added:	30		no tree isocyanate was observed by infra-red spec-
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To a 12 fiter round bottom flask equipp d with a condenser, Nitrogen flow, and temperature probe, the following materials were added:		টেডিউটের হলেও ্রেলির নারে ভারণ প্রিলেও স্থান্তর কর্মান	affected to be been been also as a
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To a 12 fiter round bottom flask equipp d with a condenser, Nitrogen flow, and temperature probe, the following materials were added:		The second secon	ta k to the same of the same o
materials were added.	55	To a 12 fiter round bottom flask equipp d with a condense	r, Nitrogen flow, and temperature probe, the following
		materials were audeu.	
		1095.1 g DGEBA countries ya selu stakemoa ou 0°9	

249.0 g

bisphenol A

	238.9 g	dodecylphenol	State Park		in the garder street of	in sur in	
	79.4 g	xylene			was law mast	\$ 5	
	The mixtur	e was heated with stirrin	ig to 120°C and vacuum	distilled by vac	uum into a Dean Sta	k tran to rem	ove
	any moistu	re. After heating to 125	oC, the following additi	on was made:	va taya am	Kilonia di La	0.0
5	3.1 g	penzyl dimethyl amine					
		erming to 152°C, the m	nixture cooled to 140°C	at which time t	the following was ad-	ded:	
	1.7 g	enzyl dimethyl amine					
		e was maintained at 13	0°C and titrated for en	oxy content at	30 minute intervals t	o an endnoin	nt of
	870 g N.V>	resin/ eq. epoxide. At	this point the following	was added	o i desembre un	o ansonapon	ir Oi
10	34.7 g	butyl cellosolve	,		. He Karaci	7 1 10 10 10 10 10 10 10 10 10 10 10 10 1	
	182.3 g	sec-butanol			មាន ប្រជាបក មេដ		
	124.3 g	diethanol amine			Pacy - Star Will		
	The mixtur	e was cooled to 90°C o	ver a one hour period a	t which time th	e following was adde	reten kina sa sa sa	
	177.7 g	ethoxylated phenolic	plasticizer		Arms 6 Ark		
15	128.2 g		•		salow besty and		£.
	48.7 g	propylene glycol phe	envl ether	trensio	Mow across from		•
	The mixture	was further cooled to		at which time th	e following was add	adt. r 1 22	
	34.8 g	limethyl amino propyl a	mine		shiriy saixa ayyan	69.713	
		was maintained 30 mir		ed to 90°C and	maintained for one h	our Thamilyt	
20	was cooled	to 68°C and blended w	ith the following until h	omodeneons.	Terror barragan	- 16.80	uie
٠.	740.3 g	component 8 10 800	on leave e do preside	i skudici mon	SOO K. HAYHI PRIVIS		
	647.7 g	component 9	•		- It is it is a second to the second	- 31 CC	
	8.9 g	anti-crater additive				.794 3, 3 4	
	The following	ng was added in order a	and vigorously mixed:		the concept 13:	roati kari sa i	
25	105.0 g	lactic acid (88%)	and vigorously militari		White the control of	1212 (1217)	
$\mathcal{H}^{\alpha}.$	√ 2034.0 g	deionized water	unabita - iliaso pattu	morfew the contracts	destimated hada	aratha a a T	
	Once homo	geneous, the mixture v	vas reduced with the a	dition of the fo	flowing over a 90 mi	nute period w	vith
	continued a	gitation:			erdozofieo lytuc		71411
	3060.2 g	deionized water			Teach Desincies	2 C(6)	
30	The above	emulsion was split into p	ortions and heated to 5	60°C and vacuu			:n:
	vents. Disti	llation was continued u	intil a concentration of	sec-butanol <	0.5% was achieved	e grilliod wo. enebàa≏allA	ate
	removed in	the process were reple	nished with deionized v	water.			410
		•	•		GAYINE TONEY.		
	Preparation	for component 11:		SWT SME THE	Manager of the left	4. 其形。	
35		อาดาษาจายนักล่า หกับว่า	e) assolvaci n 7115 pr	indic en earlige	2.2-edoug (2-mod	0 1.A5	٠,٠
	To a 12 liter	round bottom flask equi	pped with a condenser.	Nitrogen flow:	and temperature prof	e the followi	ina
463	materials w	ere added: ਿ⊽ve 🗸 🗦 🗥	rušia isti ivili Lapatina	0 5.85 HAR MC	JE L. Lebure Lew Mr.	the state of the second	9
	2343.6 g	DGEBA A STA	it will of a linear	TW - 3018/2 .35	okusa male i dina	dada tens	
	408.2 g	Dodecylphenol			to keepen visit	6. 3. 78.	
40	710.6 g	bisphenol A	wet 25 emailty.	e ion anni easy	region e consiste en	world enf	
•	178.8 g	xylene at his balls and	·衛士 5.500 可由E-MOSM	o otro a session	untimental accept	6.2.4	
· 5	The compor	ents were heated with	mixing to 120°C at whi	ch time the foll	owing was added: 3	ew xuda9	
	3.4 g tr	iphenyl phosphine	_	. ,		the federal	
	The mixture	exothermed to a peak t	emperature of 176°C at	ter which the te	mperature was main	tained/at/150	°C
4 5	for one hour	. At this time the follow	ing was added:				- Da
	2103.6 g	diepoxide of polyprop	ylene oxide (EEW=378	3 a/ea.)	After actionisms, a	anne der F	
	876.8 g	butyl cellosolve		3 - 47	MALE TO THE COMMENT OF THE COMMENT OF THE	1.47.11	
	The mixture	was cooled to 78°C at	which time the following	ig was added:	ME - CHADE VICTORIAN A	50 J 10 10 3 10 10 10 10 10 10 10 10 10 10 10 10 10	
	240.0 g	amino ethoxy ethano			direct above arew		
50	The mixture	exothermed to 97°C or	ver 30 minutes at which			oface :	S
	190.6₂g	dimethylaminopropyla			t su enol		
		exothermed to 120°C		which the tem	erature was held at	110°C for fo	ner.
	hours. After	cooling the mixture to	100°C the following wa	s added	The second of the second of the second	. 110 O 101 IC	,ui
		butyl cellosolve	:= :=::5:::15 WG		ការ មុខសាសមាន ។ ការ មុខសាសសមាន ។ គឺ		
55		peneous, the following v	was added over 25 min				A.J
	187.3 g	glacial ac tic acid	entrol de la certaine	eren. Sin eman gara site	or fell in the design of the con-	in the second of	•
	-				in a constant of the first term of the constant of the constan		
	1164.8 g	deionized water	s in Home & Dreat of	Liferon sen des	on and the factor of the second of the secon	ra emina. Managana	
	1164.8 g After one ho	deionized water ur mixing, the material	in thomas of Defect of	indoor say, no	LIME FORESTALING	er eestews	he

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Preparation for microparticle dispersion 15: To a 1 lit is round bottom flask equipped with mixing paddle, condenses and temperature probe, the following materials were add d: 217.9 g comp nent 13 Transfer to the state 78.5 a component 14 at their one The components were mixed until homogeneous. This mixture was dispersed with agitation during the addition of the following over a 15 minute periods and section is a second section and weather and section of the 544.3 q deionized water Britan & Bright & Manager and Ministry The dispersion was heated to 80°C and maintained at that temperature for:4.5 hours with continued mixing. tern traditional appropriate terms, in amost of the believe of the first of the star of the color of the first Preparation for component 16: The Free Milliam Court for the pottonic of the profession To a 5 liter round bottom flask equipped with mixing paddle, condenser and temperature probe, the following materials were added under hitrogen atmosphere: (gallari de la bendia de la discussión de progressión de la desperanda de la 458.5 g butyl cellosolve 65.4 g deionized water To an addition tank the following materials were added and mixed: A Section 1990 (1990). The control of the con 423.5 g t taene naara 385.2 git day nebutyllacrylate made on which has a fill atmenupara in all albertanisms, interpreparation of 399.2 g hydroxy ethyl acrylate hydroxy ethyl acrylate hydroxy ethyl acrylate hydroxy ethyl acrylate dimethylamino ethyl methacrylate 292.1 a 33.1 g methyl isobutyl ketone letter his 12.3 g methyl isobutyl ketone letter his 14.5 g.m. The flask was heated to reflux at 103°C at which time the nitrogen flow was discontinued. The mixture in the addition tank was added at a constant rate over two hours. The following solvent was introduced to the flask after flushing the pump and lines. Reflux was maintained for an additional 1.25 hours. 75.0 g butyl cellosolve 18 3 SEE GE 7 6 4 CELO The following initiator solution was introduced over 20 minutes: 6.6 g 2,2-azobis(2-methylbutane nitrile) dissolved in 20.0 g methyl isobutyl ketorie වඩ අයුතුය. Reflux was maintained for 2 additional hours at 105 - 110°C. The resin was cooled to 50°C and blended with the following: 111.6 g glacial acetic acid Preparation for component 17: To a 1 liter round bottom flask-equipped-with mixing paddle, condenser and temperature probe, the following 35 materials were added under nitrogen atmosphere: 346.7 g diglycidyl ether of bisphenol A 153.2 g bisphenol A 26.3 g xvlene The mixture was heated to 110°C at which time the following was added: 0.3 g triphenyl phosphine Heat was discontinued at 135°C at which point an exotherm was noted. The temperature was allowed to climb to 164°C after which it dropped to and was maintained at 150°C for a period of one hour from the point of peak exotherm temperature. The resin was cooled to 130° C at which time the following was added: butyl cellosolve Preparation for microparticle dispersion 18: 50 To a 5 liter round bottom flask equipped with mixing paddle, condenser and temperature probe, the following materials were added: 545.5 g component 16 499.4 g component 17 The components were mixed until homog nous. This mixture was dispersed with agitation during the addition of th following over a 15 minute period: deionized water The dispersion was heated to 80°C and maintained at that temperature for 4.5 hours with continued mixing.

Preparation for microparticle dispersion 19:

To a 5 liter round bottom flask equipped with mixing paddle, condenser and temperature probe, the following materials were add d:

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- 463.1 g component 16
 - 199.5 g component 14
- was 200:9 ignore. Component 1/6. In 1985 were not only out the by the profession of the excession objects in the The components were mixed until homogeneous. This mixture was dispersed with agitation during the addition of the following over a 15 minute period:
- 2136.0 g i podeionized water i en i magneti inal se benombre i base shib to bolke i and independence. The dispersion was heated to 80°C and maintained at that temperature for 4.5 hours with continued mixing.

Preparation for microparticle dispsersion 20:

- and the round bottom that backgood a framing andies, aboréans a sud-conversion a laboration for the To a 5 liter round bottom flask equipped with mixing paddle, condenser and temperature probe, the following avioreiran iça et materials were added:
 - 463.1 g component 16
 - 199.5 g component 14
 - 185.2 g component 9
- The components were mixed until homogeneous. This mixture was dispersed with agitation during the addition. $p \in \mathbb{R}^{n}$ Maly or Territary souther of the following over a 15 minute period: J. 1. 190

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deionized water 2152.0 g

The dispersion was heated to 80°C and maintained at that temperature for 4.5 hours with continued mixing. and a smithercharacteristics of the microparticle dispersions 15, 48, 19, and 20 are set forth below in Table 1.

TABLE 1

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30%	Wto & Styrene and St	29≈3/ 0 ot.	28#21 Was 1.	28429 Sole	
	Wt % n-BA	26.8	25.7	25.7	25.7 (table 4.7)
	Wt % HEA	27.7	26.6	26.628 4% 3	26.6
	Wt % DMAEMA	16.2	19.5	19.5	19.5
	Wt % VAZO 67®	2	2.6	2.6	2.6/ 000000
35	Wt/amine	978 g/eq.	817 g/eq.	817 g/eq.	817 g/eq.
2 Mary			er and a second	er sang renderar nada Lista kalandar da	hat he area alean
-	Epoxy Block			r and a a	
	Wt./epoxy	503 g/eq.	995 g/eq.	503 g/eg.	503 g/eq.
40					ensity, or the
	Stoichiometry	Cycle Wiredow	dotaca, es		
	eq.'s 3° amine	1	1		Agreement of the St.
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	Final Dispersion			۶۵۰ و	Salval notific
	Rxn % completion	94.7	81.1	80.9	77.4
	% N.V.	20.53	20.79	20.62	20.57
5 0 ty to	p.s. (nm)	201	430	197	187
46	Mw	gel	gel	gel	gel
	Wt % Acrylic	70	50.3	50.1	50.1
	₩t % Epoxy	30	49.7	24.9	24.9
4521	Wt & Crosslinker	<u> </u>	, iO	25	25
55	Wt./quat	1709 g/eq	2003 g/eq	2019 g/eq	2019 g/eq

strong test and the above oned a that respond to the following wavecommuned mixing.

Preparation for Electrodeposition Bath 21 (Comparison):

An electrodeposition bath was prepared in a 1 gallon plastic pail from the following:

2225 g Component #3

170 g Component #7

2005 g Deionized water

Preparation for Electrodeposition Bath 22 (Invention):

An electrodeposition bath was prepared in a 1 gallon plastic pail from the following:

2090 g Component #3

226 g Component #19

170 g Component #7

1913 g Deionized water

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Preparation for Electrodeposition Bath 23 (Invention):

An electrodaposition battrwas prepared in a Argallon plastic pail-from the following: Name 18 18 1994 (6)

2090 g Component (3) Behave the street is street after the self-discrete and sharing self-to has been

20 227 g Component #20

170 g Component #7

1912 g Deionized water

Preparation for Electrodeposition Bath 24 (Comparison):

An electrodeposition bath was prepared in a 1 gallon plastic pail from the following:

2298 g Component #10

386 g Component #12

1716 g Deionized water

Preparation for Electrodeposition Bath 25 (Invention):

An electrodeposition bath was prepared in a 1 gallon plastic pail from the following:

2160 g Component #10

207 g Component #18

386 g Component #12***

1646 g Deionized water

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Electrodeposition baths #24 and #25 were aged and ultrafiltered to a conductivity of 1400 micro mhos. Bare cold rolled steel and phosphated cold rolled steel panels were coated from each bath at a film build of 0.9 mils.

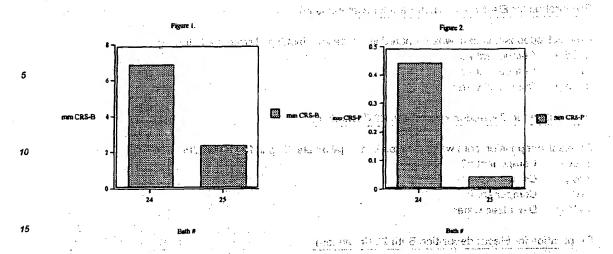
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A number of accelerated corrosion tests were carried out on the panels to characterize edge protection as well as overall corrosion resistance. As shown by figures (1) and (2) edge protection as reflected by 20 cycles SCAB testing was improved through incorporation of microparticle dispersion #18 on both substrates. Figure (1) shows edge creep over bare cold rolled steel and figure (2) illustrates the same over phosphated cold rolled steel.

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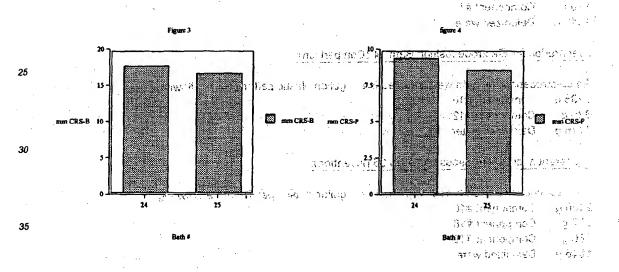
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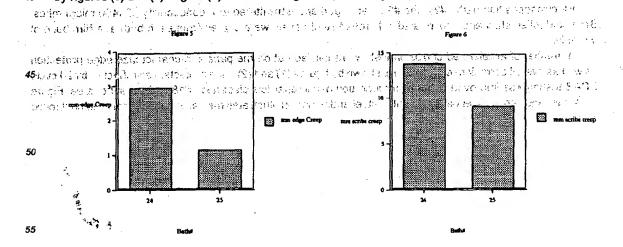


Moreover, as shown by figures (3) and (4), overall corrosion protection as reflected by creep from a scribe down the face of the panel is not compromised for either bare or phosphate-treated scell substrates.

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Similar trends are observed from 360 hours of salt spray exposure on bare cold rolled steel panels as illustrated by figures (5) and (6). Figure (5) illustrates edge creep while figure (6) illustrates creep from a center scribe.

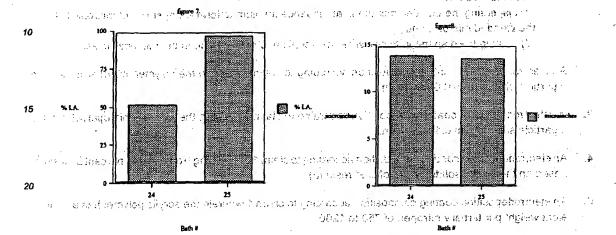


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based upon electrical resistance of a film at the edge. Isolation ability values may range from 0% (unprotected) to 100% (protected). As shown by figure (7), isolation ability is increased which reflects improved edge coverage through incorporation of microparticle dispersion:#18. As a part of the content of the content

Measured film smoothness is not adversely affected by incorporation of the invention as shown in figure (8).

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Procedure 2:

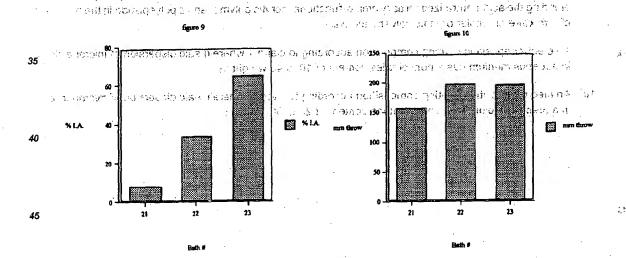
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Phosphated cold rolled steel panelsiwere coated at 275 V to a buildiof 0.4 mils from electrodeposition baths #21, #22, and #23. Incorporation of microparticle dispersions # 19 and #20 were found to improve isolation ability as illustrated in figure (9).



Ford throwpower boxes were constructed from phosphated cold rolled steel and coated at 275V to a build of 0.5 mils from electrodeposition baths #21, #22, and #23. The total distance of painted substrat was measured for each bath. The average distance of four panels are shown in figure (10). As demonstrated, significant improvements in throwing potential are achieved through incorporation of additives such as microparticle dispersions # 19 and #20.

The invention has been described in detail with reference to preferred embodiments thereof. It should be understood, however, that variations and modifications can be made within the spirit and scope of the invention.

Claims

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- 1. An electrodeposition coating composition comprising in the specific management of the specific mana
 - (a) an aqueous dispersion of a water-dispersible; electrically-depositable; film-forming; at least partially neutralized cationic resin, and

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- (b) crosslinked polymer microparticles, said polymer particles being prepared by:
 - (1) blending an acid-neutralized tertiary amino-functional acrylic polymer and a polyepoxide to form a blended mixture,
 - (2) dispersing the blended mixture in an aqueous medium to form a dispersion of microparticles of the blended mixture, and
 - (3) heating the dispersion to crosslink the acrylic and polyepoxide in the microparticles.
- 2. An electrodeposition coating composition according to claim 1 wherein the polymer microparticles have a particle size of from 0.01 to 10 µm.
- 3. An electrodeposition coating composition according to claim 1 wherein the polymer microparticles have a particle size of from 0.1 to 0.5 µm.
- 4. An electrodeposition coating composition according to claim 1 comprising from 10 to 40 percent by weight, based on the on the solids weight of said resin (a).
- An electrodeposition coating composition according to claim 1 wherein the acrylic polymer has an equivalent weight per tertiary nitrogen of 750 to 1200.
- 6. An electrodeposition coating composition according to claim 1 wherein said blended mixture further comprises a blocked polyisocyanate crosslinking agent.
- 7. An electrodeposition coating composition according to claim 1 wherein said blending step comprises and a subhending the acid-neutralized tertiary arbino-functional acrylic polymerand-applyepoxide in the presence core as of applications solvent and the acid-neutralized tertiary arbino-functional acrylic polymerand-applyepoxide in the presence core as of applications and the acid-neutralized tertiary arbino-functional acrylic polymerand-applyepoxide in the presence core as of applications and the acid-neutralized tertiary arbino-functional acrylic polymerand-applyepoxide in the presence core as of applications are according to claim 1 wherein said blending step comprises and acrylic polymerand-applyepoxide in the presence core as of a polymerand-applyers.
 - 8. An electrodeposition coating composition according to claim 1 wherein said blending step comprises blending the acid-neutralized tertiary amino-functional acrylic polymer and a polyepoxide in the presence of a mixture of a polar organic solvent and water.

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- 9. An electrodeposition coating composition according to claim 1 wherein said dispersion of microparticles in aqueous medium has a nonvolattles content of 10 to 40 weight %.
 - 10. An electrodeposition coating composition according to claim 1 wherein said dispersion of microparticles in aqueous medium has a nonvolatiles content of 20 to 30 weight %.

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EUROPEAN SEARCH REPORT

Application Number

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